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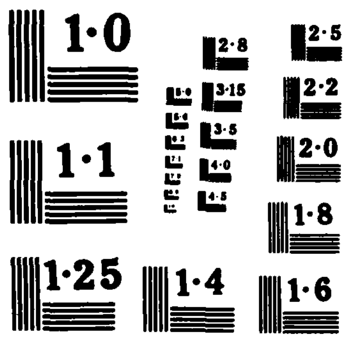

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by

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A liquid nitrogen cooled sample stage for use with the CAMECA IMS-3f ion microscope has been designed, built and incorporated into the existing laboratory instrumentation for SIMS analysis of samples at cryogenic temperatures. This is particularly useful for the ion microscopic analysis of frozen hydrated samples (biologicals), and the study of sputter and ion yields of materials in a water matrix. The application to semiconductor analysis is being explored.

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CRYOGENIC SAMPLE STAGE FOR THE CAMECA IMS-3f ION MICROSCOPE

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INTRODUCTION

The ion microscope, based on secondary ion mass spectrometry (SIMS) is a valuable tool for studying the lateral and in-depth elemental (isotopic) distributions in solid samples. To date, analyses were performed on samples that were compatible with the high vacuum environment of the instrument's sample chamber at the ambient temperature of ~25°C. This has precluded studies of trace and major elements in group II-VI compound matrices due to their high vapor pressure, and elements in a water matrix, for example.

To this end it was necessary to undertake instrumental research and to design and construct a versatile sample stage completely compatible with standard (room temperature) operations, but with cryogenic capabilities that would allow for the analysis of specialized samples.

MATERIALS AND METHODS

The main features of the standard commercial sample stage can be seen in Fig. 1 and consist of the sample holder, the sample holder mount and the sample stem. The sample holder is a stainless steel piece which serves as a holder for specimens to be analyzed, and facilitates the mechanical transfer of specimens to the sample chamber where it slides onto the mount. The sample holder mount is a stainless steel piece that holds the sample holder during analysis. This mount is fixed to the sample stage, which is attached to the backplate of the sample chamber door and contains a ceramic piece which serves to electrically isolate the sample from ground. This is important because the sample is biased at $\mp$4500 volts with respect to ground depending on the desired polarity of the secondary ions.

The cold stage had to fulfill the following requirements: 1) To preserve the fine mechanical allignment of the stage with respect to the immersion lens thus allowing the correct secondary ion optical para- meters needed by the system. 2) To enable the electrical $\mp$4500 volt potential to be maintained at the sample holder while all parts outside vacuum and in contact with the body of the instrument to remain at ground potential. 3) To achieve sample temperatures below -150°C during analysis and to maintain it with minimum fluctuation without altering the normal operation of the ion microscope.

The cold stage was therefore designed as shown in Figure 2. It consists primarily of three parts: the bellows arrangement, the cold stem and the liquid nitrogen feedthrough. The first enables the sample stem to fit to the instrument and allows some movement of the stage in the Z- direction. Next is the cold stem, which consists of a copper top used to insure good thermal conduction silver soldered to a ceramic vacuum break.[3] And finally, liquid nitrogen flows through the tube assembly of the sample stem and is in contact with the back side of the copper top.

The sample to be analyzed is held in a special sample holder designed to maximize thermal contact with the cold stage. A temperature sensor is located near the sample for accuracy.

DISCUSSION

The cold stage presented in this work was designed and tested at both room and cryogenic temperatures on the Cameca IMS-3f ion microscope. It is readily adaptable to the existing instrument and is constructed of easily obtainable parts. The stage permits holding a sample specimen at -182°C with liquid nitrogen cooling. Data were obtained for both biological specimens and the copper grid generally used for instrumental alignment under cryogenic conditions. The cold stage presents no more difficulty in operation than the standard (room-temperature) sample stage on the ion microscope.

In addition to biological studies we are exploring other uses for this cold stage on our SIMS instrument, such as ion mobility and diffusion studies, analysis of group II-VI semiconductor matrices, and other applications to the material sciences.

FIGURES

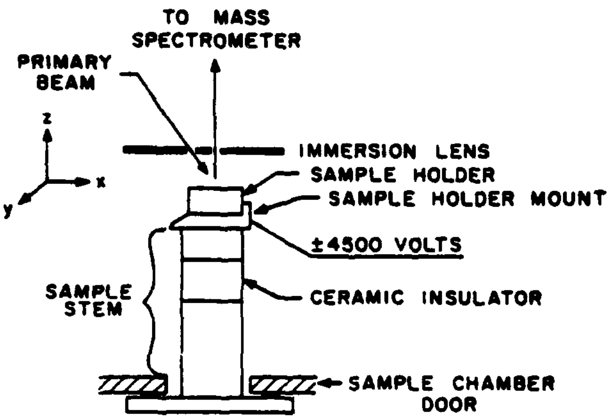

FIG. 1. Schematic of the sample stage system. Shown are the principal areas of interest, the relation of the sample to the immersion lens, and the primary and secondary beam trajectories required for analysis.

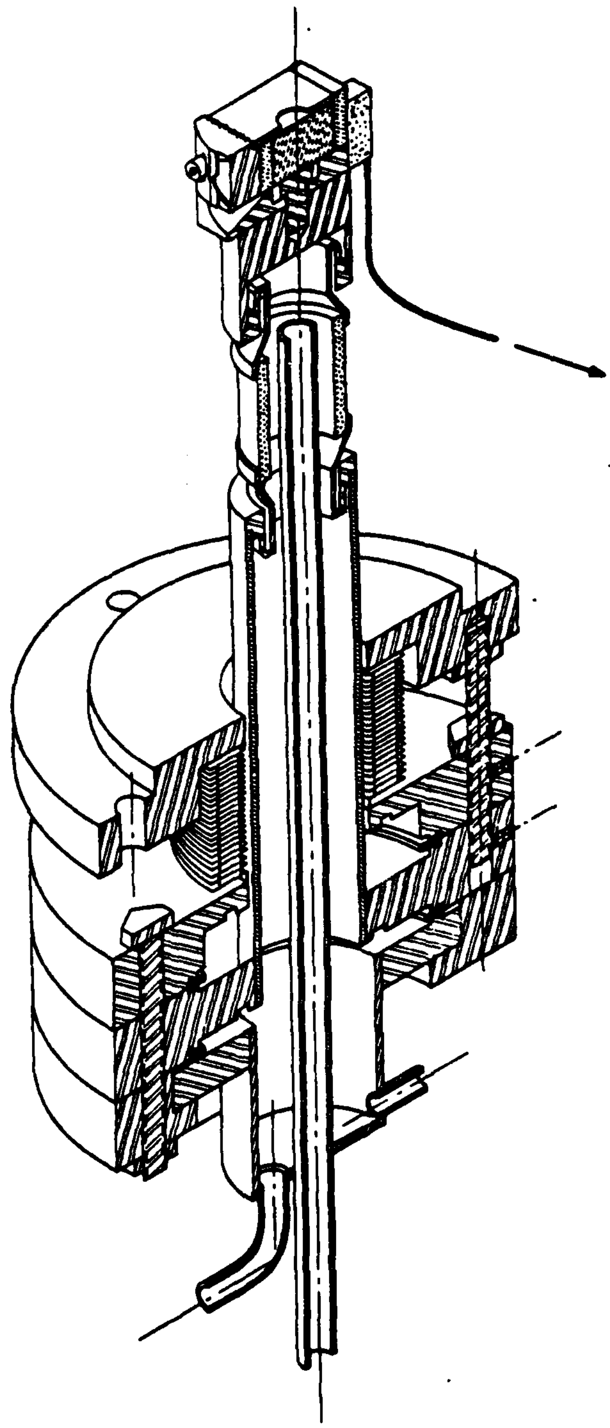

FIG. 2. Isometric-section diagram of the cold stage. Temperature sensor wire is seen leaving the system at the top right (arrow).

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